



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the application of

STEVEN LUO,  
KOJI MASAKI, and  
TATSURO HAMADA

Serial No. 09/923,983

Filed August 7, 2001

For STABILIZING HIGH-VINYL  
POLYBUTADIENE

)  
) Group Art Unit 1713  
)

) Rip A. Lee, Examiner  
)

**Certificate of Mailing**

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) 20231 on Feb 5, 2003.  
)

*Kimberly A. Bright*  
Kimberly A. Bright, Secy. to Arthur M. Reginski

**DECLARATION OF DR. STEVEN LUO**

**37 C.F.R. § 1.132**

Sir,

As the inventor of the subject application, I hereby declare that:

1. I am a resident of Akron, Ohio and am the inventor of the subject application.
2. My educational background includes B.S., Chemistry, Sun Yatsen University, 1982; M.S., M. Phil., Chemistry, Yale University, 1988; and, Ph.D., Chemistry, Yale University, 1990. I am a named inventor in at least 23 United States letters patents.
3. I have been employed by Bridgestone/Firestone Inc. for approximately seven years and am presently a Senior Research Scientist, at their Akron, Ohio research facility.
4. I understand that the claims of the present invention have been rejected because the Examiner believes it would have been obvious to combine the teachings of U.S. Patent No. 3,935,160 to Kline with those of my previous patent (U.S. Patent No. 6,117,956) to arrive at the claimed invention. I believe that the Examiner has failed to recognize that the art of choosing a suitable antioxidant for a particular polymer is largely unpredictable and empirical. Despite the teachings of Kline, I do not believe that

one skilled in the art can predict that the same dialkylaminomethyl phenols taught by Kline would also be effective for syndiotactic 1,2-polybutadiene.

5. In support of my beliefs, I offer the following.

6. As part of development work associated with the claimed technology, I have discovered that 2,4,6-tris(dimethylaminomethyl)phenol, which contains three dialkylaminomethyl functional groups, is ineffective in preventing syndiotactic 1,2-polybutadiene from thermal crosslinking. This discovery was observed in the following comparative experiment.

7. An oven-dried 1-liter glass bottle was capped with a self-sealing rubber liner and a perforated metal cap. After the bottle was thoroughly purged with a stream of dry nitrogen gas, the bottle was charged with 105 g of hexanes and 228 g of a 1,3-butadiene/hexanes blend containing 21.9% by weight of 1,3-butadiene. The following catalyst ingredients were then added to the bottle in the following order: (1) 0.050 mmol of iron(III) 2-ethylhexanoate, (2) 0.20 mmol of bis(2-ethylhexyl) hydrogen phosphite, and (3) 0.70 mmol of tri-n-butylaluminum. The bottle was tumbled for 1.5 hr hours in a water bath maintained at 80°C. The polymerization was terminated by adding 1 mL of isopropanol to the polymerization mixture. 1.5 g of 2,6-di-t-butyl-4-(dimethylaminomethyl)phenol (Ethanox 703) in 7 mL of toluene was then added to the polymer cement as the stabilizer. The polymerization mixture was shaken at 80°C for 10 minutes and cooled to room temperature. The mixture was dried under vacuum at 40°C, giving 50.7 g of syndiotactic 1,2-polybutadiene containing the stabilizer. The yield of the polymer is 49.2 g (98.4%) with the stabilizer excluded. The polymer had a melting temperature of 147°C, a 1,2-linkage content of 89.7%, a syndiotacticity of 81.6%, a number average molecular weight of 161,000, a weight average molecular weight of 340,000, and a polydispersity index of 2.1.

In order to demonstrate the effectiveness of 2,6-di-t-butyl-4-(dimethylaminomethyl)phenol in preventing the thermal crosslinking of syndiotactic 1,2-polybutadiene, about 5 g of the polymer was pressed for 5 minutes at 210 °C to form a film with a thickness of about 0.5 mm. The film was tested for solubility in 1,2-

dichlorobenzene. The film was found to be completely soluble in 1,2-dichlorobenzene at 130°C, which indicated that the film contained no gel. Therefore, thermal crosslinking or gelation of syndiotactic 1,2-polybutadiene had been prevented by the presence of 2,6-di-*t*-butyl-4-(dimethylaminomethyl)phenol as the stabilizer. ✓

8. The procedure described in Paragraph 7 was repeated except that 2,6-di-*t*-butyl-4-(dimethylaminomethyl)phenol was replaced by 2,4,6-tris(dimethylaminomethyl)phenol. The syndiotactic 1,2-polybutadiene film which was formed by pressing for 5 minutes at 210°C, was completely insoluble in 1,2-dichlorobenzene, which indicated that the polymer had been completely gelled. Therefore, I concluded that 2,4,6-tris(dimethylaminomethyl)phenol was ineffective in preventing the thermal crosslinking or gelation of syndiotactic 1,2-polybutadiene. ✓

9. I declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true and, further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the U.S. Code and that such willful false statements may jeopardize the validity of this application and any patent issuing thereon.

Respectfully submitted,



Steven Luo, Ph.D.

Date: February 5, 2003

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) \_\_\_\_\_  
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**DECLARATION OF DR. STEVEN LUO**  
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Steven Luo, Ph.D.

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